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BINDER LEACHING OF NATURALLY HYDRAULIC LIME MORTARS

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ABSTRACT

Masonry built in lime mortar is one of the principle building materials used in traditional construction. This form of construction can convey the appearance of strength and stability and could be argued to epitomise built homogeneity. This may however not reflect reality due to deterioration of the wall core resulting in an alteration in the effective thickness. Predicted future climate change models for northern regions, indicate more instances of heavy rainfall. This will undoubtedly result in materials used in mass masonry structures becoming saturated for extended periods, increasing instances of binder migration (binder leaching). The leaching process involves the deterioration of binder structure via dissolution and removal of calcium into the pore fluid. This can, in many cases, result in reprecipitation of calcium on the external skin of the structure. The effect of this phenomenon on the physical properties and performance of lime mortar has not yet been fully investigated but early stage results indicate a reduction in mechanical strength and an alteration of the materials moisture handling characteristics. The current EPSRC funded research project (EP/G064865/1) aims to ascertain the extent of binder leaching by inducing an artificially accelerated leaching environment. Leaching trials have been carried out on a variety of natural hydraulic limes (NHL), both carbonated and uncarbonated, representing those potentially found on mass masonry structures. The present work aids our collective understanding of this fundamental deterioration process enabling prediction of mortar condition and performance to be achieved. Determining traditional building resilience has never been as important, especially given the broader context of climate change.

Keywords: binder, dissolution, leaching, lime, mortar

INTRODUCTION

An estimated 446,000 pre 1919 dwellings, totalling 20% of the building stock, exist in Scotland today. The vast majority of these are believed (McKinney, 2007, Urquhart, 2006) to be constructed of stone. More specifically, it has been estimated that there are some 24,000 traditional built stone facades in Glasgow alone, requiring approx £500M to repair (Urquhart, 2006). Of course, structures constructed in traditional mass masonry are not restricted to Scotland or to tenement forms, with innumerable

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low rise domestic buildings, historic bridges, viaducts, infrastructural masonry (i.e. retaining walls), castles and palaces existing within the UK and across the world. Mass masonry construction is generally composed of an internal and external skin of masonry bedded in lime mortar with a lime built rubble core. The structural performance of these walls relies upon the effective thickness and homogeneity to create stability.

Anecdotal evidence from building professionals is that situations have arisen in which the mortars have been found to be binder deficient (Forster, 2007). In many situations, voids have been found in wall cores behind the apparently sound masonry pointing and bedding mortar, and this has been subsequently associated with progressive collapse in many traditional buildings (Kent, 2005). Beckman, (1995:86) indicates that *'if the skins are thin compared to their height and if they are not well connected to the core, they may buckle'*. It is therefore clear that the condition of the core is critical for stability.

McKibbins *et al* (2006:78) believes that *'Contributory mechanisms for deterioration of masonry in all types of structures include, moisture saturation and leaching of mortar.... Leaching results in physical loss of strength and adhesion. Mortar that has undergone severe leaching can become weak and friable, and is easily lost from joints by washing-out or compressive extrusion in areas of high stress, resulting in local stress concentrations and loosening masonry units.'* Beckman (1995:86) reinforces this view highlighting the implications of long term saturation, indicating *'rainwater enters the core and dissolves or leaches out the lime and loosens the fine particles. These are then washed down, leaving behind cavities, which grow until the rubble collapses into them'*. Binder leaching is clearly a function of moisture content and it is evident that due to increased rainfall associated with climate change (Hume, 2002; Cassar & Hawkins, 2007) masonry fabric will be wetter, for longer. Certain components within the mortar may dissolve and move from the body of the material and be re-deposited within the pores, construction voids, or may be left simply upon the wall faces of the building (see plate 1).

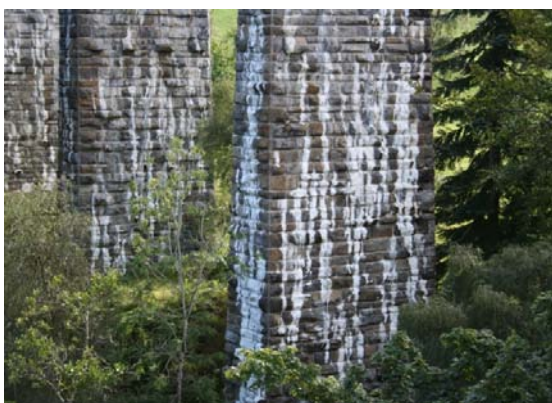


Plate 1: Lime leachate to piers

The most vulnerable binder components to dissolution are (Hewlett, 1998, Taylor, 1990) calcium hydroxide ($\text{Ca}(\text{OH})_2$) and calcite (calcium carbonate, CaCO_3). The hydraulicity of the original mortars will have a significant influence upon the set characteristics of the material. Non-hydraulic and feebly hydraulic limes require a high degree of carbonation for their set propagation and have a correspondingly low

proportion of hydraulic components within their composition. Intuitively, these forms of lime binders are the most susceptible to dissolution, when placed in saturated, cold conditions and is a function of the relative proportion of $\text{Ca}(\text{OH})_2$.

The extent to which binder dissolution occurs has yet to be fully undertaken. It is however, assumed that the rate of deterioration will be extremely slow and may take decades rather than years. Although these time frames may be very slow, it must be emphasised that many of the structures that we are dealing with may already be many decades old, and those that are considered historic in nature may be centuries old. Those buildings that are ruinous may have been roofless for several hundred years, further exacerbating the problem. This Engineering & Physical Science Research Council (EPSRC) funded project (EP/G064865/1) has undertaken to evaluate this phenomenon and this work establishes some of the preliminary findings.

RESEARCH OBJECTIVES

The aim of the research is to establish the rate and consequence of soluble binder leaching from various lime mortar types commonly used in traditional mass masonry construction. It determines the effect on physical and chemical properties of the mortars, and specifically the work investigates the changes to the macro and micro pore structure and its effect on moisture transfer mechanisms. The project objectives are:

- 1) Determine the rate of free lime binder leaching ($\text{Ca}(\text{OH})_2$) in a range of hydraulic lime mortars, using ammonium nitrate accelerated deterioration method.
- 2) Assess the changes in moisture handling characteristics of the samples via sorptivity testing.
- 3) Assess the effect of binder loss upon the compressive and flexural strength characteristics of the lime mortars.
- 4) Characterise the change in the composition of the mortars.
- 5) Develop a predictive model for assessing the influence of binder depletion on compressive and flexural strength of the mortars to give building professionals and engineers a rational basis for of in-service performance prediction.

EXPERIMENTAL PROCEDURE

Sample manufacture and curing

A range of lime binders adopting a constant binder to aggregate ratio for all samples of 1:3 (by weight) was used. This regime was a modification of BS EN 459-2 (2010) and BS EN 1015 (2002). Well graded siliceous sand (Cloddach concrete sand) ensured isolation of variables as it does not contain calcinitic mineral components. This aggregate is representative of materials commonly used for the repair of traditional masonry structures in Scotland. St Astier natural hydraulic lime (NHL) was used as it is the most commonly adopted binder for conservation works in the UK. NHL2 (feebly hydraulic), NHL3.5 (moderately hydraulic) and NHL5 (eminently hydraulic) samples were manufactured and cast into 160 x 40 x 40mm prisms moulds, following BS 459:2 (2010). These samples were cured in a TAS series 3 environmental cabinet under two different regimes in order to modify the physical and chemical characteristics of the mortar; one ensured a full carbonation set (60% RH at 20°C) whilst the other un-carbonated (100% RH at 20°C). This was an attempt to simulate conditions that may be encountered in existing structures, namely, fully carbonated mortars located in the outer masonry and partially carbonated or

uncarbonated material within the wall cores and inner sections of bridge construction. The carbonation regime followed that established by Lawrence (2006).

Accelerated leaching

A chemical extraction process using ammonium nitrate (NH_4NO_3) was utilised to accelerate the leaching of calcium (Ca^{2+}) ions from the NHL samples. Nguyen *et al*, (2007) formerly used this technique to evaluate deterioration in cements and concretes and indicated an accelerated leaching of Ca^{2+} ions in the region of 200-300 times that that of the natural phenomena. Modification of Nguyen *et al*, (2007), experimental procedure was undertaken to take account of the different characteristics of naturally hydraulic lime mortar compared to the relatively more durable concretes and ordinary Portland cements. This resulted in a dilution of the ammonium nitrate concentration reducing the solutions aggressivity. Samples were immersed in a solution of 1 mol/l (1M) concentration of ammonium nitrate in deionised water. The volume of the solution was kept constant and monitoring of the pH and temperature was continuously logged via a data acquisition system (see fig. 1).

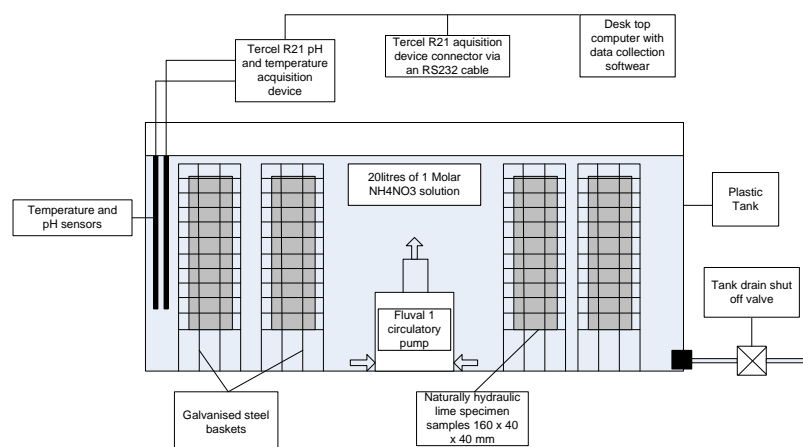


Fig 1: Ammonium nitrate extraction apparatus (after Nguyen, 2007)

A pump ensured that the ammonium nitrate solution was circulating, thereby preventing non uniform solution concentration. The samples were immersed for 1, 4, 9, 16, 25, 36, 100 and 169 days. Additional samples were ran in parallel for the initial 36 days. These were kept in vats of de-ionised water in an attempt to establish a datum for the natural dissolution rate.

Physical and chemical investigation of pre and post accelerated samples

A range of tests were established to determine the physical and chemical characteristics of the materials. Control samples were used to provide a baseline for pre deteriorated properties. Samples that had been subjected to accelerated deterioration were tested at various intervals (as previously outlined) adopting the following techniques:

i) Phenolphthalein testing: In order to measure the speed of dissolution the samples that were subject to the ammonium nitrate and deionised water were fractured and surfaces were sprayed with phenolphthalein solution. This was undertaken to measure, and ultimately calculate the leaching depth of the $\text{Ca}(\text{OH})_2$ from the samples (see plate 2). **ii) Sorptivity testing:** The sorptivity of the mortar samples were evaluated using the ‘sharp front theory’ established by Hall & Hoff, (2002:97). The sorptivity of each prism sample type was determined by immersing the bottom surface

of the samples in water and measuring weight gain / over-time following the basic function $t^{1/2}$ (see fig.4). This gave an indication of the change in pore structure and subsequently the moisture transfer characteristics. **iii) Flexural and compressive strength:** Each sample type was tested in a Lloyds universal testing machine (model M5K) at specific intervals. This enabled an assessment of the influence of loss of $\text{Ca}(\text{OH})_2$ on the physical characteristics of the mortar samples. **iv) Petrographic analysis:** A selected range of mortar samples were assessed under polarised light microscopy, enabling the pore structure and mineral composition of the binder to be determined by point counting. This was undertaken to identify the influence of leaching of $\text{Ca}(\text{OH})_2$ and to evaluate the remaining components, such as calcite and calcium silicate hydrates (C-S-H). **v) ESEM analysis and EDAX:** The freshly fractured surfaces were studied adopting the Philips XL30 ESEM, whilst simultaneously undertaking Energy Dispersive X-Ray Micro-analysis (EDAX) techniques to determine the chemical composition of the products of hydration of the binders used, and the surface characteristics of the material.

Preliminary results and discussion

Provisional experimental results for the ammonium nitrate (NH_4NO_3) extraction tests will be discussed. It must be emphasised that the project is still ongoing and these results simply establish early stage findings of the physical properties, and more specifically the compressive, flexural and sorptivity values. The results focus on the NHL5 and NHL2, carbonated and un-carbonated mortar samples as they are representative of the extremes in material composition and more specifically the relative quantity of calcium hydroxide ($\text{Ca}(\text{OH})_2$) and calcite (CaCO_3) if carbonated.

Compressive and flexural results

The test results indicate a clear difference between the samples subjected to the NH_4NO_3 and the de-ionised water (see fig 2 & 3). The samples generally correlate with perceived wisdom, in so much as, the samples with a higher proportion of hydraulic set and therefore a reduced carbonation requirement (such as an NHL5 samples) have a smaller decline in strength.

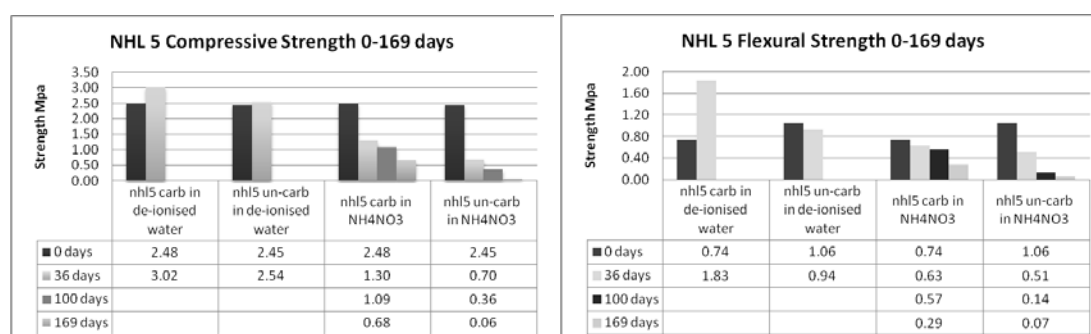


Fig. 2. Compressive and flexural strength changes over time NHL5

The NHL5 carbonated samples in the de-ionised water increased in compressive and flexural strength over the 36 day period while the un-carbonated samples showed an increase in compressive strength and a marginal decline in their flexural strength (See fig. 2). This may be associated with favourable hydration curing environment (i.e. submerged in water). The NHL5 samples in the NH_4NO_3 , both carbonated and un-carbonated samples exhibited a large decrease in their compressive and flexural strength over the same time frame, with compressive strength being more effected.

After this initial 36 day period the NHL5 carbonated samples did not exhibit a large decline in compressive and flexural strength (100 and 169 day testing period). In comparison the un-carbonated sample continued to decline in strength, with the result that there was negligible strength left at the 169 day period (See fig. 2).

The NHL2 samples in comparison to the NHL5 reflect a different pattern. All the NHL2 samples decreased in both compressive and flexural strength when subjected to the de-ionised water and the NH_4NO_3 . The small loss of strength in the carbonated NHL2 relative to the NHL5 samples subjected to the NH_4NO_3 could potentially be attributed to the high proportion of calcite formation. Conversely, the slow hydration of belite (C_2S) [the main hydraulic component in hydraulic limes] and its subsequent conversion into C-S-H in the NHL5 samples would make the carbonation formation disproportionately important in terms of strength development. The un-carbonated NHL2 samples in the NH_4NO_3 had insignificant strength left after the initial 36 day period (See fig. 3). This would generally be associated with higher proportions of $\text{Ca}(\text{OH})_2$ and calcite leaching from the samples.

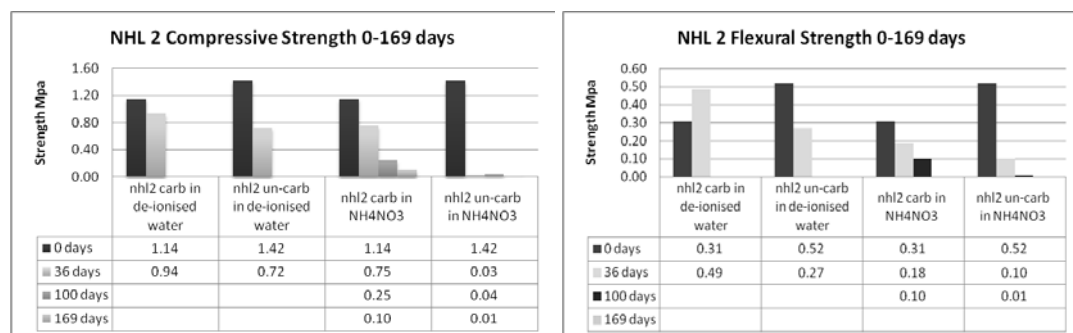


Fig. 3. Compressive and flexural strength changes over time NHL2

Sorptivity: The sorptivity values for the un-carbonated samples were lower than the carbonated ones. This was unexpected with the NHL5 carbonated samples attaining the highest values of all. The NHL5 samples, subjected to the de-ionised water exhibited the highest reductions in their sorptivity values (see fig 4). The carbonated NHL5 also exhibited a reduction after 36 days in the NH_4NO_3 whereas the un-carbonated samples increased. (See fig.4)

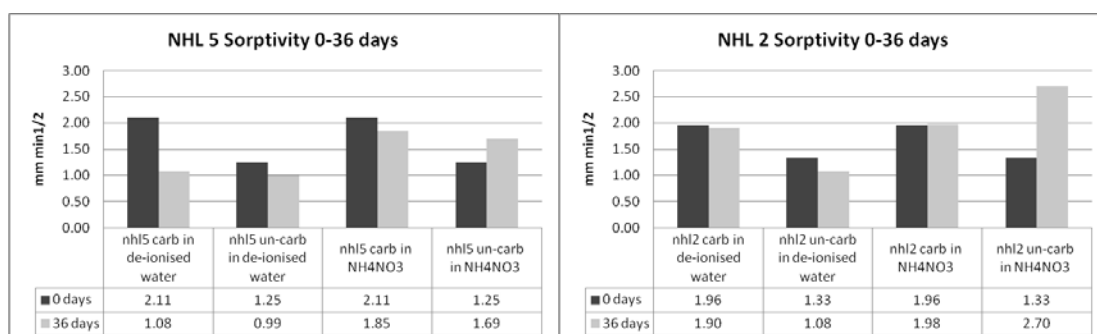


Fig. 4. Change in sorptivity value of NHL2 and NHL5

The carbonated NHL2 sorptivity values were not greatly affected over the course of the 36 days test runs, whereas the un-carbonated NHL2 samples reduced significantly in strength and exhibited a 50% increase in their sorptivity value. This is indicative of

washout of the $\text{Ca}(\text{OH})_2$ and is visually represented in the phenolphthalein staining test results seen in plate 2.

Dissolution front assessment (phenolphthalein staining): It was observed during the first experimental run that all the un-carbonated $[\text{Ca}(\text{OH})_2]$ lime material had leached out within the first 36 days. As such it was not possible to track this dissolution front. Attempts to ascertain the extent to which the NH_4NO_3 accelerates the process and subsequently effects the materials physical and chemical properties were undertaken.

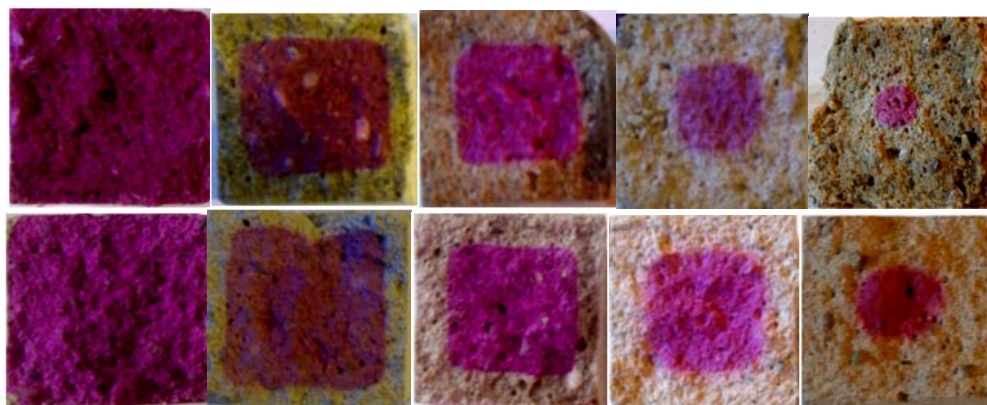


Plate 2: Phenolphthalein test results on uncarbonated samples subjected to NH_4NO_3 at 0-4-9-16-25 days (top NHL2, bottom NHL5)

The second test run measured the dissolution front by spraying the samples with phenolphthalein solution at designated time intervals, measuring the distance from the outside, to where the boundary of $\text{Ca}(\text{OH})_2$ material remained. This is shown as the dark regions in plate 2 and reflects an alkalinity pH 8.6-12. The un-carbonated NHL2 samples leached $\text{Ca}(\text{OH})_2$ at an accelerated rate compared to the un-carbonated NHL5. The advancing dissolution front correlates with the physical testing results attained over the initial 36 days. The increase in the depth of dissolution front of the $\text{Ca}(\text{OH})_2$ can be directly related to an increase in sorptivity, and a decrease in the compressive and flexural strengths. The increased rate with which the NHL2 samples leached can be attributed to the quantity of $\text{Ca}(\text{OH})_2$ contained within the material. It is reasonable to assume that this situation leads to a greater proportion of the composition of the material being lost via dissolution as the front advances through the sample; increasing the pore size, and pore interconnectivity.

CONCLUSIONS

These results confirm that the ammonium nitrate extraction method accelerates the leaching of $\text{Ca}(\text{OH})_2$, or un-carbonated components of the mortar samples. This is evident by the phenolphthalein test results that clearly show the leaching front of the un-carbonated components and complete $\text{Ca}(\text{OH})_2$ loss was noted by 36 days. This is important as it highlights the vulnerability of un-carbonated lime to saturated environments especially over longer time frames.

This information has practical implications for the performance of lime mortars and will have the greatest influence on binders containing a higher proportion of $\text{Ca}(\text{OH})_2$, such as NHL2. Freshly placed lime mortars have been shown to have a greater resistance to $\text{Ca}(\text{OH})_2$ loss than would have been initially expected. This will go some

way to alleviate performance fears of lime mortars that are inadvertently saturated. Practically, this preliminary study illustrates the significant alteration in the materials properties. It is evident that long term saturation of masonry and associated mortars should be considered when evaluating traditional and mass masonry structures. The loss of binder results in a reduction in both compressive and flexural strength and can also be correlated with an alteration in the moisture transfer properties of the samples. These characteristics are important to attain a better understanding of mortar performance but should be seen within the context of an early stage development of a wider range of tests. When combined these should enable a rigorous method for predicting the impact of the physical performance of binder loss to be achieved.

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